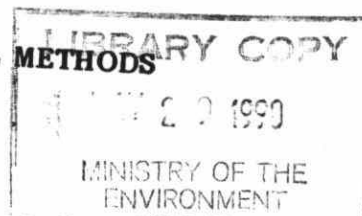


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PART D

ANALYTICAL METHODS



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A COMPARISON OF MASS SPECTRAL INSTRUMENTAL CAPABILITIES  
(LRMS, HRMS & MS-MS) FOR CHLORINATED DIBENZO-P-DIOXIN AND  
DIBENZOFURAN DETERMINATION  
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Ultra-trace determinations of chlorinated dibenzo-p-dioxins (CDDs) and dibenzofurans (CDFs) in environmental samples are performed using the technique of gas chromatography - mass spectrometry (GC-MS). Many different GC-MS techniques have been employed, including the use of various types of mass analyzers. The three analyzers that have been most extensively investigated include low resolution quadrupole (GC-LRMS) systems; high resolution, double-focussing systems (GC-HRMS); and tandem mass spectrometer systems (GC-MS-MS) based upon a dual quadrupole and collision cell (CAD cell) design.

The capabilities of the various GC-MS systems available for CDD/CDF determination are not the same. However, there is no single system that is best suited for all applications. The advantages and disadvantages of each type of system are summarized in Table 1. Previous studies have shown some of the differences that exist between certain instrument types for the analysis of real samples (1,2). In a joint study carried out by the Ministry of the Environment and SCIEX a comparison of GC-LRMS and GC-MS-MS analysis of fly-ash was made. It was shown that interferences detected by GC-LRMS were not detected in the GC-MS-MS data and it was also noted that the quantitative results obtained by GC-MS were consistently higher than those derived by

GC-MS-MS (1). Recently Clement et al (2) compared the instrument sensitivities of GC-LRMS, GC-HRMS and GC-MS-MS and found the HRMS and MS-MS techniques produced comparable sensitivities for the determination of 2,3,7,8-TCDD, while all three techniques were comparable for the determination of OCDD. (See Table 2)

To date, no systematic comparison of the capabilities of GC-LRMS, GC-HRMS and GC-MS-MS for CDD/CDF determinations has been reported. Such studies are needed to define the best area of application for each technique. The study to be presented will provide a comparison of the capabilities of each instrument type for the determination of CDD/CDF's as well as determine the linear dynamic range and the detection limits of each instrument type. The instrumental systems to be used during this study include a Finnigan 4500 (LRMS); a Hewlett Packard MSD (LRMS); a VG-ZAB-HS double focusing instrument (HRMS) and a Finnigan TSQ-70 MS-MS system.

During the initial stages of this study CDD and CDF standards at varying concentrations will be analyzed using selected ion monitoring to determine instrument linearity as well as detection limits. A comparison of real sample analysis will be carried out on fly-ash, in which fly-ash extracts at varying dilutions will be analyzed by each instrument type. The flyash extract will be prepared and diluted in a manner so as to represent detected levels of CDD/CDF above the detection limit, at the detection limit and below the detection limit of a LRMS instrument. A comparison of detected values, response factors and the presence or

absence of interferences will be made between each instrument type.

Preliminary work has indicated a marked difference in detection limits of different mass spectrometer techniques (Table 2). It has also been observed that the operating modes of certain instrument types affects the results obtained from CDD/CDF analysis (3). This phenomenon will also be investigated in this study.

TABLE 1: COMPARISON OF RELATIVE CAPABILITIES OF GC-LRMS, GC-HRMS AND GC-MS-MS

FEATURE	GC-LRMS	GC-HRMS	GC-MS-MS
SELECTIVITY	LOW	HIGH	MEDIUM
SENSITIVITY	MEDIUM	HIGH	HIGH
SAMPLE THROUGHPUT	HIGH	LOW	HIGH
UNIQUE FEATURES	---	EXACT MASS DETERMINATION	SPECIAL SCAN MODES
OPERATOR SKILL	LOW	HIGH	MEDIUM
COST OF EQUIPMENT	LOW	HIGH	HIGH

TABLE 2: RELATIVE SENSITIVITIES OF INSTRUMENTAL TECHNIQUES FOR CDD/CDF (2)

	AMOUNT INJECTED	OBSERVED SIGNAL : NOISE		
		LRMS	HRMS	MS-MS
2,3,7,8-TCDD	12 pg	10:1	125:1	100:1
OCDD	24 pg	15:1	25:1	15:1

References:

- (1) Shushan, B., Tanner, S.D., Clement, R.E., & Bobbie, B., Chemosphere 55, 14, 843-846 (1985)
- (2) Clement, R.E., Bobbie, B., & Taguchi, V., Chemosphere, 15, 1147-1156 (1986)
- (3) Ontario Ministry of the Environment, unpublished data.



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